LASER REFLECTANCE INTERFEROMETRY SYSTEM WITH A 405 NM LASER DIODE FOR IN SITU MEASUREMENTS OF CVD DIAMOND THICKNESS

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Abstract

In situ monitoring of the thickness of thin diamond films during technological processes is important because it allows better control of deposition time and deeper understanding of deposition kinetics. One of the widely used techniques is laser reflectance interferometry (LRI) which enables non-contact measurement during CVD deposition. The authors have built a novel LRI system with a 405 nm laser diode which achieves better resolution compared to the systems based on He-Ne lasers, as reported so far. The system was used for *in situ* monitoring of thin, microcrystalline diamond films deposited on silicon substrate in PA-CVD processes. The thickness of each film was measured by stylus profilometry and spectral reflectance analysis as a reference. The system setup and interferometric signal processing are also presented for evaluating the system parameters, i.e. measurement uncertainty, resolution and the range of measurable film thickness.

Keywords: interferometry, thin films, CVD, diamond, *in situ* measurements

1. Introduction

The thickness measurement in thin dielectric films manufactured by PA-CVD processes is critical for the evaluation and optimization of a technological process [1-3]. It allows determining the film growth rate and deposition of films with a specified thickness. Among numerous thickness measurement methods, the most often used ones are (I) stylus profilometry [4], (II) scanning electron microscopy (SEM) [5] and (III) optical methods (ellipsometry, time- and spectral-domain interferometry) [6-13].

The optical methods are generally characterized by an indirect way of measurement. Film thickness is being determined based on parameters of light transmitted or reflected from the film surface: (I) polarisation (ellipsometry) [8], (II) intensity (time-domain interferometry) [10-13] or (III) spectrum (spectral-domain interferometry) [9,13]. Optical techniques are based on the assumed film optical model therefore the model parameters inaccuracy can cause errors in the thickness measurement. This is the main drawback of the optical methods compared to the direct measurements by stylus profilometry or SEM [9].

However, the optical methods allow performing a no-contact and non-destructive measurement. Moreover, they enable *in situ* film monitoring which is critical in diagnostics or studying of CVD process kinetics [6,14]. In optical systems, the angle of incidence of light onto the film may be fixed [10-12] or scanned within a specified range [15-17]. The second approach allows an analysis of additional features of the film, such as surface quality [15] or mechanical stress [16], however they are difficult to apply for *in situ* measurement, where the light angle of incidence is defined by the construction of the CVD reactor chamber.

Laser reflectance interferometry (LRI) is an *in situ* monitoring technique for growing diamond films in CVD processes [10-12]. It involves the measurement of the intensity of laser light reflected from the film during its growth. Several LRI systems based on He-Ne lasers have been reported [10-12]. Film thickness can be calculated by fitting the parameters

of the assumed film optical model (thickness, refractive index and surface roughness) to the measured interferometric signal [10] or by counting the extremes of film reflectance [12].

The extremes counting methods allow to perform the thickness measurement with a resolution equal to a quarter of wavelength of the light in the investigated film [12]. He-Ne lasers with a wavelength of 632.8 nm provides the measurement resolution of 65.6 nm (with refractive index of diamond equal to 2.41 [12]). The aforementioned value is valid assuming normal incidence of laser light on the measured sample; it decreases with decreasing angle of incidence.

Currently, the progress in laser diodes technology makes it possible to build inexpensive LRI systems based on light sources with light wavelengths shorter than those in He-Ne lasers. This enables achieving a better measurement resolution. The authors have built a novel LRI system with a 405 nm laser diode. In this system, the layer thickness is calculated from the number of extremes in the intensity of light reflected from the growing diamond film. The system setup, applied algorithms of the digital signal processing and evaluation of system metrological parameters are presented below.

2. Measurement principles

2.1. Measurement setup

The 405 nm LRI home-made setup is presented in Fig. 1. It consists of a 405 nm laser diode (L) with 100 mW output power. Laser radiation is transmitted onto the sample (S) inside the CVD reactor chamber via a quartz viewport at the angle of incidence of 45°. The light reflected from the diamond film is transmitted via a second viewport to a silicon photodiode (D). The generated photo-current is transformed to a voltage signal by a transconductance amplifier (W), converted to digital form by an analog-to-digital converter (A) and sent to a PC.
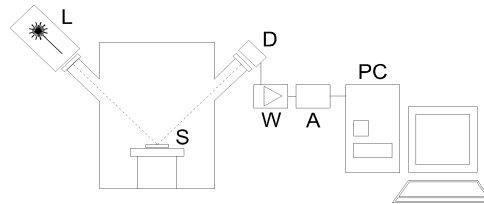

Fig. 1. 405 nm LRI system for measuring the growth of thin diamond films.

2.2. Interference in thin dielectric films

The reflectance of a thin dielectric film, deposited on a specified substrate, can be calculated according to the following formula:

$$R = \frac{R_1 + R_2 + 2\sqrt{R_1 R_2}\cos(\beta d)}{1 + R_1 R_2 + 2\sqrt{R_1 R_2}\cos(\beta d)}, \tag{1}$$

where R is the reflectance of the film; R_1 and R_2 are reflection coefficients at the ambient-film and film-substrate boundary, respectively; d is the thin film thickness; and β is given by (2):

$$\beta = \frac{4\pi}{\lambda} n_1 \cos\phi_1, \tag{2}$$

where λ is the wavelength of light; n_1 is the refractive index of the film and ϕ_1 is the angle of refraction of light inside the film.

Assuming a 405 nm light source, 45° angle of light incidence and the refractive index of diamond equal to 2.46 (value for natural diamond at a wavelength of 405 nm), the value of β for the designed system can be calculated from (3):

$$\beta = 0.0732 \ nm^{-1} \ . \tag{3}$$

During the film growth, as the film thickness increases, the signal detected by the photodiode achieves its extremes for:

$$\beta d = m\pi \ , \tag{4}$$

$$\text{for} \quad m = 0,1,2,.... \ . \tag{5}$$

The signal calculated from (1) for increasing values of d and light polarized perpedicularly to the plane of incidence is presented in Fig. 2. In practice, roughness of the film surface introduces scattering of the incident light. It results in a drop of the amplitude of oscillation in reflectance. The experimental interference signal is presented in Fig. 3.
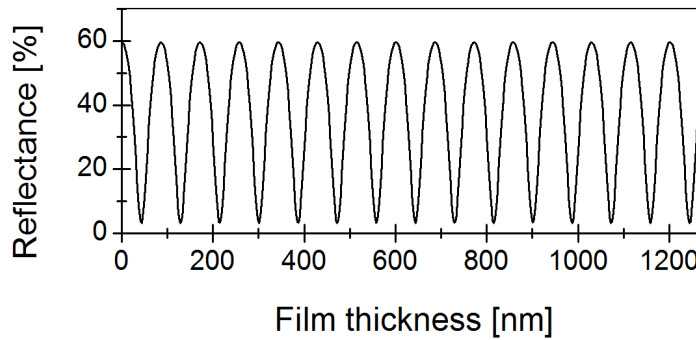

Fig. 2. Theoretical reflectance of an ideal thin diamond film deposited on silicon substrate. The distance between subsequent maxima is equal to 85.8 nm, which is twice the resolution of film thickness measurement, based on extremes counting method.
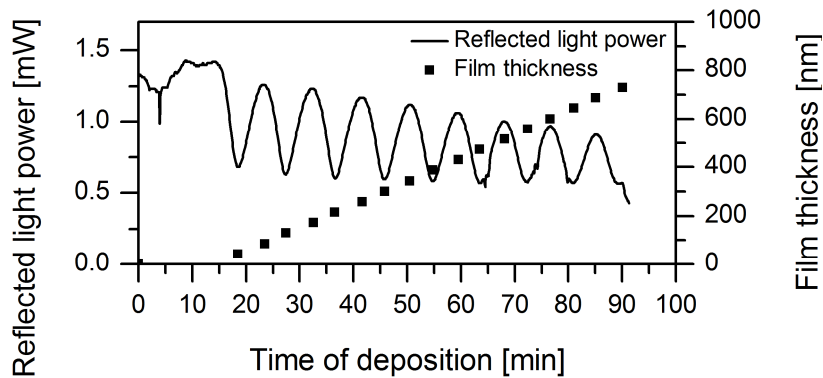

Fig. 3. Experimental interferometric signal recorded during growth of the CVD diamond and calculated film thickness. A drop in the amplitude of oscillation caused by increasing roughness of the film surface can be observed. The measured power does not drop below 0.5 mW due to additional light emission by plasma inside the reactor.

2.3. Signal processing

The signal processing algorithm is presented in Fig. 4. It is based on detection of extremes in the recorded signal. First, in order to eliminate false extremes caused by noise, the signal is filtered using a low-pass digital filter. Such elimination is necessary for fully automatic measurement, where each false extreme would falsely increase the total calculated film thickness. The following procedure of fitting the filter parameters has been used.

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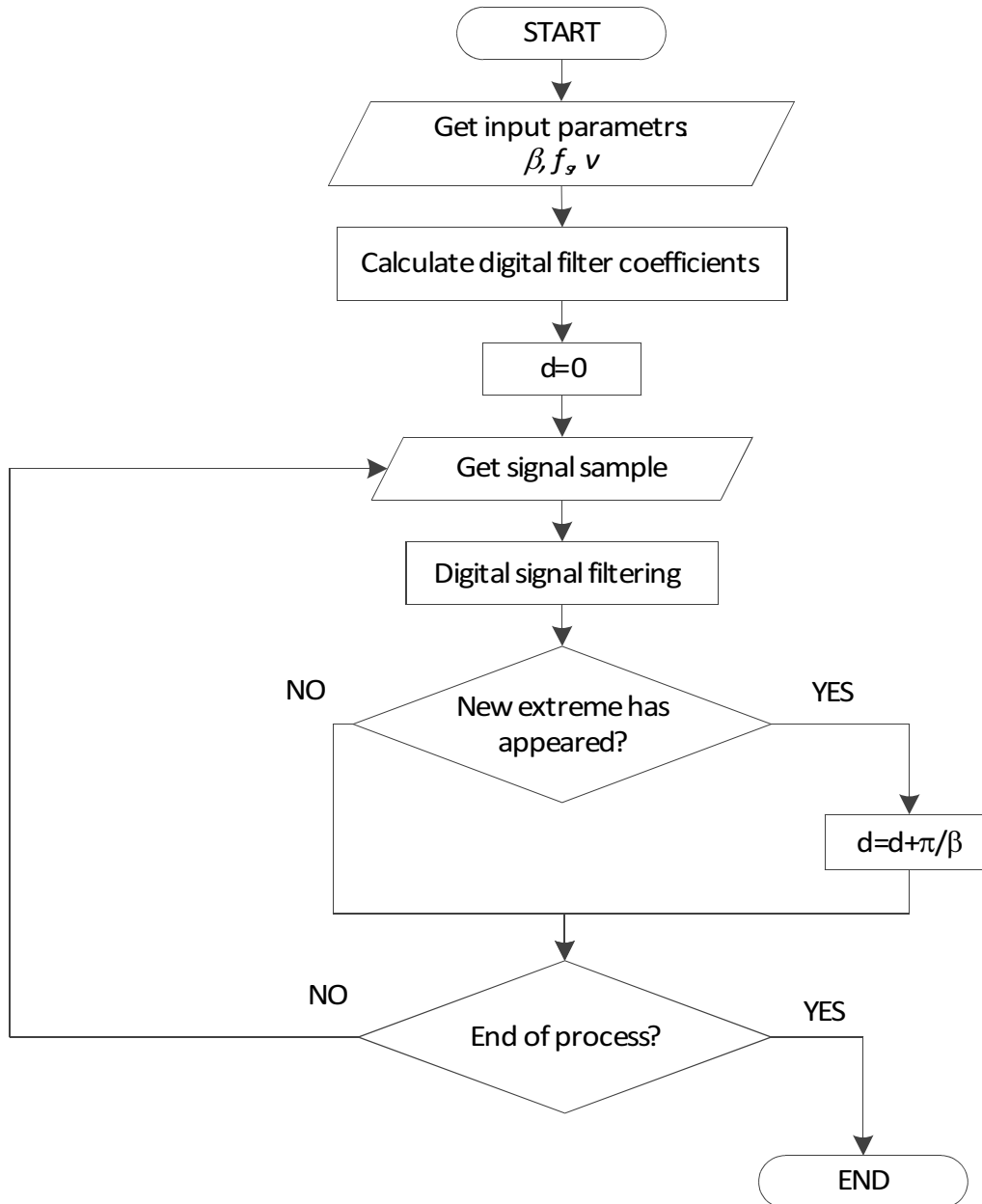
Fig. 4. Algorithm for calculating the diamond film thickness from 405 nm LRI signal.

The predicted, normalized frequency of oscillation is calculated based on the knowledge of β, the signal sampling frequency (f_s) and a rough estimate of the film growth rate (v), as (6):

$$\frac{f}{f_s} = \frac{\beta v}{f_s}.$$
(6)

The IIR Butterworth filter coefficients are calculated using the bilinear transformation method with cut-off frequency equal to 1.5 of the normalized frequency calculated using (6).

The thickness of the film is calculated based on the number of extremes in the filtered signal, according to (7):

$$d = m\frac{\pi}{\beta},$$
(7)

where m is the number of recorded extremes.

3. Analysis of system performance

3.1. Theoretical analysis

According to (1) and (2), the following effects can influence the 405 nm LRI system error:
- a shift in the refractive index of the film by δn [18]
- a quantization error arising from the fact that the number of extremes can only assume positive integer values
- inhomogeneous film growth with varying film thickness for different spots on the film, marked as δd.

Assuming the lack of correlation between the above listed effects, the total relative measurement uncertainty can be estimated as:

$$\varepsilon = \sqrt{\left(\frac{\delta n}{n}\right)^2 + \left(\frac{\delta d}{d}\right)^2 + \left(\frac{\delta m}{m}\right)^2}.$$
(8)

Measurement resolution is equal to film growth corresponding to two neighboring extremes in the interference signal. Its value can be calculated based on (3) and (4). The least detectable difference Δd of film thickness is given as (9):

$$\Delta d = \frac{\pi}{\beta} = 42.9 \, nm.$$
(9)

Only the δm value in (8) can be calculated analytically; it can reach a maximal value of 1. To achieve a relative quantization error less than 10%, more than 10 reflectance extremes need to be recorded. This requires that the grown diamond films are at least 429 nm thick. The values of δn and δd can be estimated by *ex situ* examination of diamond films with spectral interferometry.

Higher measurement resolution could be obtained by advanced interference signal processing algorithms. Such methods are efficient for flat surfaces deposited with atomic precision. CVD diamond films exhibit significant surface roughness that introduces light scattering and deterioration of the interference signal. This makes advanced interference signal processing difficult to apply.

The trends in roughness and refractive index were taken from our previous studies based on Raman spectroscopy and AFM/SEM measurements [19, 20]. Deconvolution of Raman

spectra gives the sp^3/sp^2 ratio which can be used for refractive index trend estimation, while AFM/SEM results in grain size distribution supply information about the roughness variation. These results were specifically used during the LRI setup and algorithm design.

In the case of polycrystalline CVD diamond films, the thickness measurement resolution of 43 nm is sufficient to effectively monitor the growth.

3.2. Experimental measurements

The system performance was evaluated by the following procedure. A series of CVD diamond film growth experiments with different thickness values have been performed. In each case, the films were deposited on a p-Si (111) substrate. Microwave H_2/CH_4 plasma at 50 torr of pressure and a hydrogen flow of 300 sccm and a substrate temperature of 700°C was used. Three different methane flow rates (1%, 2% and 5% of flow) were used. The processes with a specified methane flow and time of deposition are presented in Table 1.

Table 1. Parameters of CVD processes used for testing 405 nm LRI system performance.

Sample number	CH_4/H_2 flow rate, %	Time of deposition, min
1	1	126
2	5	49
3	1	120
4	2	93
5	1	197
6	2	135
7	5	99

The thickness of each film was measured *in situ* using the designed system. In order to obtain reference thickness values, the deposited films were studied mechanically by stylus profilometry and optically by spectral-domain interferometry. The substrate was masked during deposition in order to create the film edge for profilometrical studies using a profilometer with an accuracy of 5 nm. It was observed that the mask application causes disturbance in the film growth. This resulted in a change of film thickness in the proximity of the mask. This change manifests itself as interference fringes visible in the range of 2-3 mm from the film edge.

The range of measured film profile from the mask edge was set high enough to reach beyond the area in which the presence of mask influenced the film growth. However, the disturbance in film growth caused by the mechanical mask renders stylus profilometry inaccurate for examining the thickness of a thin diamond film.

The spectral interferometry method involved the measurement of film spectral reflectance (with normal light incidence) and evaluation of the film parameters (thickness and surface roughness) by fitting theoretical spectra to the measured ones.

Reflectance spectra were measured at the central point of each sample. A tungsten halogen light source (BPS101, B&W TEK Inc., USA) and a fibre-optic spectrometer (USB4000, Ocean Optics, USA) were used in this procedure. Each measurement was

preceded by recording the spectrum of light reflected from bulk, monocrystalline Si (111) with a known reflectance spectrum for the wavelength range of 400-900 nm.

Theoretical spectra were calculated using the theory of light reflection from rough surfaces [21-22].

Variations in the reflectance spectrum of films with an optical thickness shift of 1 nm can be easily detected. However, deviations of the refractive index limit the accuracy of the spectral interferometry method. As described in section 3.2., refractive index fluctuations of 5% should be expected. Using optimization methods, such as the Levenberg-Marquadt algorithm [23] allows finding an approximated value of the film refractive index from the reflectance spectra.

In situ measurements and the reference thickness values obtained by both aforementioned methods are presented in Table 2, and graphically in Fig. 5. For all optical measurements, the film refractive index and dispersion have been assumed to be equal to those of natural diamond. Relative errors of LRI measurement, compared to each reference method has been presented as well.

Relative differences between results of profilometry and LRI vary strongly for different samples, but smaller errors have been generally obtained for thicker films. It suggests that they are mostly caused by quantization error, as described further. Differences between LRI and spectral reflectance measurement methods take the same sign for all samples and very similar values for all of them. This suggests the presence of systematic error in one of the methods.

Table 2. Comparison of the film thickness measurements by LRI and reference methods.

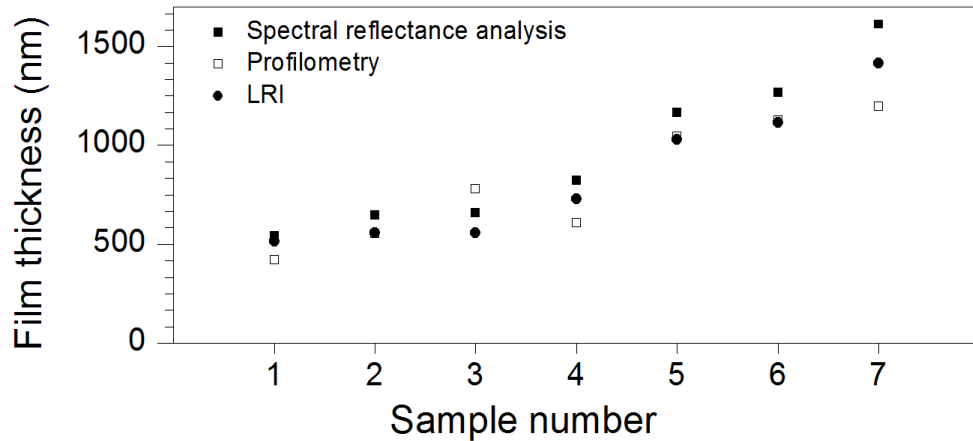| Sample number	Thickness measured by LRI [nm]	Thickness measured by profilometry [nm]	Relative error of LRI	Thickness measured by fitting spectral reflectance [nm]	Relative error of LRI
1	515.0	423.4	22%	542	-5%
2	557.9	552.3	1%	638	-13%
3	557.9	780.1	-28%	659	-15%
4	729.6	607.6	20%	823	-11%
5	1030.0	1043.9	-1%	1164	-12%
6	1115.9	1125.5	-1%	1266	-12%
7	1416.3	1197.3	18%	1613	-12%

Fig. 5. Comparison of film thickness measured by using the 405 nm LRI system and reference methods.

The results in Fig. 5 and Table 2 show that the differences between *in situ* determination and the reference methods can exceed the calculated uncertainty. This finding concerns samples 1 and 3 for profilometry, and sample 3 for the spectral reflectance analysis.

Differences between LRI and profilometry results vary in character. Samples 2, 5 and 6 show very good agreement between the two methods since other LRI results assume smaller or larger values compared with profilometry data. LRI measurement requires an assumption of the film refractive index value. If its real value differs from the assumed one, erroneous results are obtained. On the other hand, masking a part of the substrate in order to prepare a sample for profilometric examination disturbs the film growth. These two phenomena are likely to be the main causes of the observed differences.

For every film, the thickness values measured by spectral reflectance analysis are about 10% greater than those obtained by LRI. This suggests a systematic phenomenon influencing the results. One possible explanation is the position of the substrate during the CVD processes. The laser beam used for *in-situ* measurement usually does not hit the exact centre of the growing film where the growth rate is expected to achieve its highest values.

3.3. Estimation of measurement uncertainty

The Cauchy model of material dispersion has been assumed for estimating the shift in refractive index of the films:

$$n = n_0 + \frac{N_2}{\lambda^2} + \frac{N_4}{\lambda^4}, \qquad (10)$$

$$k = k_0 + \frac{K_2}{\lambda^2} + \frac{K_4}{\lambda^4}, \qquad (11)$$

where n and k are real and imaginary parts of the complex refractive index. Examination of thin diamond films by spectroscopic ellipsometry shows that the Cauchy model can be successfully applied to film refractive index in the visible light range [24].

Model parameters n_0, N_2, N_4, k_0, K_2 and K_4 from (10) and (11) have been calculated by fitting theoretical reflectance spectra to the measured ones using the Levenberg-Marquadt Algorithm (LMA). LMA is an optimization algorithm which fits parameters of the theoretical model to the measured values in order to minimize the square mean error of the fitted function [23].

A successful application of LMA requires that the chosen initial values of the model parameters are close to the final ones. For initial values that are too far from the solution, the algorithm may find only a local minimum of the error.

To calculate the reflectance spectra, initial values of thickness and surface roughness (defined as standard deviation of the film thickness) are required. Efficient results of LMA calculation have been obtained using the initial value of film thickness measured by the LRI system (Table 2). The initial value of surface roughness was kept at 0. The natural diamond refractive index was chosen for initial Cauchy model purposes.

The calculated values of *n* for a wavelength of 405 nm are presented in Table 3.

Table 3. Refractive indices of deposited films calculated by fitting spectral reflectance method.

Sample number	Film refractive index
1	2.39
2	2.46
3	2.55
4	2.36
5	2.43
6	2.41
7	2.57

The mean value of the obtained refractive indices is equal to:

$$\bar{n} = 2.45, \tag{12}$$

which is close to the assumed value for natural diamond. The standard deviation of the results and its value relative to the mean are equal to, respectively:

$$\sigma_n = 0.080, \tag{13}$$

$$\frac{\sigma_n}{n} = 3.3\%. \tag{14}$$

To obtain the value of uncertainty of film thickness due to inhomogeneous film growth (Δd from (8)), the thickness of each sample was measured using spectral-domain interferometry at seven different points on the film surface. For each film, the standard deviation of thickness relative to the mean was calculated. The mean relative standard deviation from all films was equal to:

$$\frac{\sigma_d}{d} = 5.0\%. \tag{15}$$

Assuming that $\Delta n = 3\sigma_n$ and $\Delta d = 3\sigma_d$, the values of relative measurement uncertainty from (8) for different thicknesses of the film were plotted in Fig. 6. It was noted that for films thicker than 500 nm, the quantization error becomes insignificant compared to the uncertainty of refractive index and the effect of inhomogeneous film growth. This finding justifies the use of the simple extremes counting method as an efficient way to calculate film thickness from the interferometric signal. For films with thickness values above 500 nm, the uncertainty

reaches values smaller than 20%. Results in Fig. 6 may explain larger differences between LRI and profilometry measurements for a film with smaller thickness, as shown in Tab. 2.
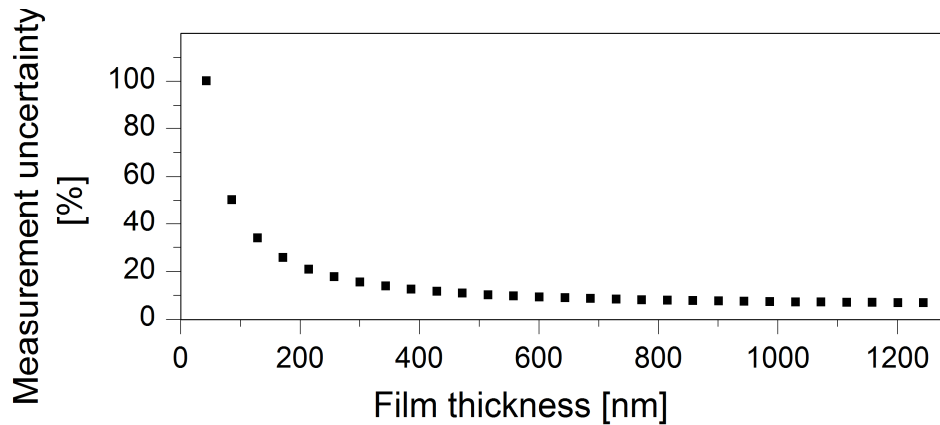

Fig. 6. Relationship between the measurement uncertainty and the layer thickness.
A decreasing influence of quantization error on the entire uncertainty value can be observed.

3.4. Influence of film surface roughness

The uneven growth of polycrystalline diamond film causes an increase of film surface roughness during CVD. Rough surface introduces the loss of coherence of the reflected light wave and increases the diffuse part of the reflectance [19]. This effect manifests itself as a drop in oscillation amplitude and the mean value in the interference signal, as presented in Fig. 3. A decrease of oscillation amplitude below the detection level makes further thickness measurements impossible. The maximal sample thickness possible to be measured depends on particular parameters of the process. For the performed test processes listed in Table 1, the upper limit of thickness measurement was estimated at 2 μm.

4. Conclusions

A novel 405 nm LRI system for *in situ* diagnostics of thin film growth has been developed. The performance of the system was studied by analyzing seven CVD diamond film samples deposited on silicon substrates. The film thickness and refractive index were examined in samples using stylus profilometry and spectral reflectance analysis.

For LRI measurements, film thickness was calculated by counting extremes in an interferometric signal. This method introduces a quantization error to the results. For suffcently thick films, the value of this error is insignificant compared to the uncertainty of the film refractive index and the effect of uneven diamond growth. The relative uncertainty of the thickness measurement was estimated to be better than 20% which is valid for a film thicker than 500 nm.

Measurement resolution for the extremes counting method was 43 nm. The upper limit of measurable film thickness is determined by the film surface roughness, which causes a drop in oscillations in the interferometric signal. The limit value was experimentally determined to be 2 μm. In specific cases, the limit value depends on the parameters of the CVD process which determines the film surface roughness.

Acknowledgements

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